

# PATENT SPECIFICATION

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## (54) HIGH-YIELD PULP SUITABLE FOR ABSORBENT PRODUCTS AND PROCESS FOR ITS MANUFACTURE

(71) We, MO OCH DOMSJÖ AKTIEBOLAG, a Swedish Body Corporate of Fack S-891, O1 Ornskoldsvik-1, Sweden, do hereby declare the invention for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

- 5 The present invention relates to a cellulose pulp having improved properties for absorp- 5  
 tion purposes and to a process for the manufacture of such pulp in a high yield using lig-  
 nocellulosic materials, such as wood, bagasse or straw, as starting materials. The pulp pro-  
 duced in accordance with the present invention is particularly well suited for the manufac-  
 10 ture of absorbent products such as various types of soft paper (tissue paper), cellulose wad,  
 10 diapers, sanitary napkins and tampons.  
 For the manufacture of such absorbent products, chemical pulps, such as sulfite or sulfate  
 pulp, are generally used at present. In the preparation of such pulps, however, less than half  
 of the wood fibre is recovered, which is a serious disadvantage in view of the increasing  
 15 shortage of fibrous raw materials. If the wood material is to be made use of in a more  
 efficient manner, the pulp yield should be increased and the fibres reclaimed. However, it 15  
 has been found difficult to reclaim sanitary paper in an economic way. Thus, in order to  
 utilize the limited supply of fibrous raw material more completely, it is possible to raise the  
 pulp yield. Conventional methods for preparing so-called high-yield pulp comprises grind-  
 20 ing logs against a rotating grinding stone or initially disintegrating the wood into smaller  
 pieces, so-called chips, and feeding them into a disc refiner having a rotating disc provided  
 with ridges or two counter-rotating ridged discs, the wood fibres being liberated during  
 passage of the wood chips between the discs. In a further development of the disc refiner  
 process, the chips are preheated with steam before they are supplied to the disc refiner, said  
 25 preheating serving to soften the wood lining to facilitate fiber liberation. The so-called  
 thermomechanical pulp obtained by this method has a higher proportion of long fibers than  
 pulps obtained by the chip refining or log grinding methods. Such processes, however, have  
 the disadvantage of a high energy consumption. It is also known to delignify the lignocellu-  
 30 losic material only in part by means of sulphur compounds at a high temperature (above  
 160°C), before subjecting it to defibration, in order to obtain a high pulp yield. By controll-  
 ing the pulping conditions it is possible in this case to obtain pulp yields which vary from 30  
 60% to 93% (so-called chemimechanical pulp). The chemimechanical pulping process has a  
 substantially lower energy consumption than that required for the preparation of mechan-  
 35 al pulps of the above types, but has the disadvantage of requiring considerable amounts of  
 sulfurous chemicals, which require an expensive chemicals recovery system. Another dis-  
 advantage of this method resides in the dissolution of large amounts of organic substances 35  
 which have to be collected and treated to avoid contamination of the environment. The  
 preparation of unbleached and bleached mechanical pulps also results in the production of  
 waste material which has to be purified before it can be discharged to the environment.  
 40 A disadvantage common to all the pulping processes above described is that the pulps  
 produced have to be screened in various expensive and bulky equipment to remove coarse  
 particles or to effect enrichment of prime quality fibers. If screening is carried out at a low  
 concentration of the fiber material, only low concentrations of chemicals and dissolved  
 organic substances are obtained in the effluent liquid, which makes chemical recovery and  
 45 purification difficult and economically unattractive.  
 A further disadvantage of the prior art high-yield pulps is that they tend to stratify during 45

shipping and storage, so that their bulk is considerably reduced.

The present invention provides a light-coloured cellulose pulp having a low resin content and high bulk, containing 0.1 to 1.0% by weight of sulfur; a proportion of retained fibers above 25% by weight on a 20 mesh screen (ASTM E 11-61), as determined by a fiber fractionation analysis (as hereinafter defined) using a Bauer-McNett apparatus; a proportion of soft, sulphonated fiber bundles, each consisting of 2 to 4 individual fibers, of at least 10% by weight; a resin content of less than 0.8% by weight as determined by a dichloromethane extraction procedure according to SCAN-C7: 62 and a bulk when dried of above  $20 \times 10^{-3} \text{ m}^3/\text{kg}$ , all percentages being based on the weight of oven dried pulp.

The pulp of the present invention has been found to be markedly superior to hitherto known cellulose pulps for liquid absorption purposes, it has an improved stability to stratification (i.e. the pulp does not tend to form itself into discrete layers) and it is prepared at much lower cost on account of low wood or other lignocellulosic material consumption and low energy requirement.

The test procedure used for the fiber-fractionation analysis using the Bauer-McNett apparatus is described in SCAN-M6:69. That reference does not, however, describe the use of a 20 mesh sieve, but the procedure can be modified with respect to the sieve size as is illustrated in Pulp and Paper Magazine of Canada 69 (1968): 17, pages 69 to 73 (T331-T335) and in an article by J. E. Tasman in TAPPI (1972): 1, pages 136 to 138. The sieve cloths in the test procedure used for the present invention were:

Mesh No. ASTM E 11-61	Sieve opening mm
20	0.841
150	0.104

25	20	0.841	25
	150	0.104	

Preferably, a pulp according to the invention contains 0.2 to 0.6 % sulphur, has 30 to 65 % of its fibers retained on a 20 mesh screen (ASTM E11-61) as determined by the fiber fractionation analysis using the Bauer-McNett apparatus, contains 15 to 30 % soft, sulphonated fiber bundles each containing 2 to 4 individual fibers and has a bulk above  $25 \times 10^{-3} \text{ m}^3/\text{kg}$ .

It is particularly advantageous for the pulp according to the invention to have a pH above 6, when dried. The pH is most preferably 7 to 10, so that acidic products in the liquid to be absorbed may be neutralized, resulting in reduced irritation of skin in contact with the liquid and the absorbent product.

The present invention also provides a process of making a light-coloured cellulose pulp by washing lignocellulosic material with water, impregnating it with a digesting liquid containing sulfur dioxide-containing digesting chemicals, partly digesting the impregnated lignocellulosic material to a degree of sulfonation corresponding to 0.1 to 1.0%, preferably 0.2 to 0.6 %, by weight sulfur, based on the weight of the treated cellulose material, at a temperature of 373° to 443°K so that at the termination of the partial digestion, the digesting liquid contains no free or combined sulfur dioxide, mechanically partly defibrating and simultaneously bleaching the lignocellulosic material, the resulting defibrated pulp containing at least 10% by weight, based on the weight of oven dried pulp, of soft, sulphonated fiber bundles, each comprising 2 to 4 individual fibers.

Suitable digesting chemicals for use in the process of this invention are bisulfite and/or sulfite of sodium, potassium, ammonium, calcium or magnesium, sodium bisulfite and sodium sulfite being preferred. The amount of digesting chemicals in the digesting liquid should normally be at most equivalent to 20 grams of  $\text{SO}_2$  per liter. The pH of the digesting liquid used in the impregnation of the cellulose material is preferably from 5 to 11, more preferably from 6 to 9, and the impregnating step is suitably carried out so that the material absorbs from 0.9 to 1.5 liter of solution per kilogram of dry material. The temperature used in the digestion step may be lower than that normally used in the preparation of chemimechanical pulp to obtain partial digestion to a degree of sulfonation in accordance with the invention. The temperature range is 373° to 443°K, preferably 383° to 403°K. It is suitable to maintain the same temperatures during impregnation as in the digesting step, and it is particularly suitable to treat the water-washed material with steam prior to impregnation.

The partial digestion in the process of the present invention is essential to obtain the advantageous properties possessed by the product produced in accordance with the invention. It results in the lignin present in the lignocellulosic material being softened and partly sulphonated, so that it can be partly dissolved subsequently.

The degree of sulfonation corresponds to 0.1 to 1.0%, preferably 0.2 to 0.6 %, by weight of sulfur for example, in the form of  $\text{SO}_3^-$  and  $\text{HSO}_3^-$  ions based on the weight of the treated lignocellulosic material. Within the scope of the invention, different species of wood

will necessitate different digesting conditions to obtain the degree of sulfonation above referred to. It is essential that the digestion is carried out so that after termination of the partial digestion no free or combined sulfur dioxide is present in the digesting liquid. To this end, the residence time of the material and the temperature must be controlled accordingly. Suitable residence times in the impregnating stage are 0.5 to 5 minutes, and suitable residence times in the digesting stage are 0.5 to 30 minutes, preferably 2 to 10 minutes.

In accordance with this invention, after the partial digestion, the resulting pulp is mechanically defibrated in a defibrating device in an incomplete manner, so that the defibrated pulp will contain at least 10% of fiber bundles. The "fiber bundles" are soft fiber bundles each comprising 2 to 4 individual fibers. It is not intended to include hard fiber bundles containing unsulfonated wood and soft fiber bundles having 5 or more individual fibers, so-called shives. Preferably, the defibration is carried out in a manner such that the defibrated material will contain 15 to 30% fiber bundles of the type referred to. Furthermore, the defibration is preferably carried out so that any reduction of the fiber length during defibration is avoided, which may be achieved by treatment in high consistency mills. Carrying out the defibration in the above manner in combination with the partial digesting step, results in a final product which is especially suitable for absorption purposes, having such properties as high bulk, good water absorbing capacity, including rate of absorption and retention capacity, good resistance to stratification separation into discrete layers under storage conditions, low resin content and good brightness.

It is preferable according to the invention, that a resin-removing chemical and a bleaching chemical are present in the defibrating step. Suitable resin-removing chemicals are phosphates, complexing agents and/or surfactants, phosphates being particularly preferred, and the removal of resin is carried out in an alkaline environment, alkali metal hydroxides such as sodium hydroxide being preferred as alkali. The pH of the resin-removing liquid supplied to the defibrating device is preferably above 11 to provide an initial pH of the pulp suspension above 9, preferably above 10. Suitable bleaching chemicals to be supplied to the defibrating device together with the resin-removing chemicals are alkali metal peroxides, such as sodium peroxide, or hydrogen peroxide, but also so-called reducing bleaching agents, such as dithionites, hydroxylamine, thiourea and thioglycolic acid can be used. Other oxidizing bleaching agents, e.g. ozone, persulfates, oxygen, peracetic acid and borax, may also be used as bleaching agents according to the invention. In the defibration process, a homogeneous admixture of the added chemicals is obtained as well as a rapid and efficient saponification of fatty acids and resin acids, the latter facilitating dispersion of unsaponifiable portions of the extractive substances. The relatively high temperature and the intense mechanical working contribute to these reactions.

The partial defibration step is suitably followed by dewatering the resulting pulp suspension to a solids content of at least 30 %, preferably 45 to 55 %, at a high temperature, suitably at least 348°K. This high temperature results in the best possible separation of saponified and dispersed extractive substances and in a high solids content of the pulp to be subjected to a subsequent drying step. Also, the dewatering step results in the chemicals and dissolved organic substances to be recovered being present in a high concentration. The subsequent drying step preferably results in a solids content of the pulp of at least 86%, e.g. 88 to 92%.

An advantage resulting from the use of the present invention is that the fiber yield is very high, generally from 85 to 95%, e.g. from 90 to 94 %. Also, the process of the present invention requires a very low energy input of 200 to 1100 kWh per 1000 kg of dry cellulose fibers produced. Further, the invention has the advantage that the volume of discharged excess liquid from the process is small and concentrated with respect to impurities and that withdrawal of the liquid can be made at one point. This permits a single and economic recovery of used chemicals and/or purification of recycled process waters. Also, the equipment used is of low cost, since it is not necessary to screen the cellulose material, which moreover further reduces the energy consumption. Still another advantage of the invention is that the heat generated in the process can readily be collected and utilized. Thus, in the process, low pressure steam is generated which can be used for evaporation or remote heating purposes. An additional advantage of the invention is that a very little amount of water is needed, usually about 3 m<sup>3</sup> of water per 1000 kg of dried cellulose material produced. The corresponding amount for producing chemical pulp is about 250 m<sup>3</sup> and for producing mechanical or thermomechanical pulp about 20 m<sup>3</sup>.

Lignocellulosic materials, such as wood, bagasse or straw, are used as starting materials in the process of this invention. It is particularly suitable to use wood fibers, which also include a certain amount of recycled fibers. When fresh or stored wood is used, the logs are first cut in a chipper, whereupon the resulting chips are washed in hot water. At the same time, iron objects which may damage movable parts of the machine, are preferably removed by means of electromagnets. The defibrating device used in the process is particular-

ly sensitive to damage from such objects.

In a preferred embodiment of the invention, which can be carried out in an apparatus such as that illustrated in the Figure of the accompanying drawings, the chips are washed in the chip washer 1 and are then conveyed hot to a steaming vessel 2 wherein they are treated with saturated steam at atmospheric pressure. This step of the process is referred to as steaming and serves to drive out air from the chips and to equalize their moisture content. After steaming the chips are fed into a screw conveyor 3, which debouches into an impregnating chamber 5. Alternatively, the washed chips are introduced directly into the screw conveyor 3, i.e. with no steaming taking place. During the passage of the chips through the screw conveyor 3, they are compressed so that excess liquid flows out through a discharge outlet 4 of the screw conveyor. After the chips have passed through the screw conveyor 3, they are allowed to expand freely in the impregnating chamber 5. The impregnating chamber 5 is filled with a weak solution of sodium, potassium, ammonium, calcium or magnesium bisulfite and/or sulfite as digesting liquid. It is preferred to use sodium bisulfite or sulfite. To achieve a rapid sulfonation of the lignin during the treatment in the impregnation chamber and in the digester 10, the pH of the digesting liquid should preferably be from 5 to 11, more preferably from 6 to 9. The  $\text{SO}_2$  content of the digesting chemicals in the digesting liquid is preferably not above 20 g of  $\text{SO}_2$  per liter. It has been found that during the free expansion of the chips in the solution, the chips generally absorb from 0.9 to 1.5 liter of solution for each kilogram of dry chips. The volume of liquid absorbed varies with the species of wood. Within the impregnating chamber 5 there are two vertical screws 6, 7 which convey the chips upwardly to the top of the chamber. To achieve homogeneous impregnation and to obtain a partial sulfonation in the liquid phase, the chips are lifted slowly in the impregnating chamber. At the same time, the liquid level in the impregnating chamber is maintained as high as possible. This is effected automatically by means of sensors and control valves, which are not shown in the Figure. The treatment in the impregnating chamber is most suitably carried out at a temperature of  $373^\circ$  to  $443^\circ\text{K}$ , preferably  $383^\circ$  to  $403^\circ\text{K}$ , for a period of 0.5 to 5 minutes. From the top of the chamber, the chips drop into a digester 10 situated below the top of the impregnating chamber. The chips leaving the impregnation chamber have a solids content of about 35%.

In the digester 10 the chips are partially sulfonated at a temperature of  $373^\circ$  to  $443^\circ\text{K}$ , preferably at  $383^\circ$  to  $403^\circ\text{K}$ . These temperatures also prevail in the impregnating chamber 5, since it communicates with the digester 10. The digester is heated by saturated steam introduced through pipe connection 8, i.e. the continued sulfonation of the wood takes place in the vapor phase. The residence time of the chips in the digester is normally 0.5 to 30 minutes. A residence time of 2 to 10 minutes has been found particularly suitable. In accordance with the invention, the residence time and the temperature are controlled so that no free or combined  $\text{SO}_2$  can be detected in the digestion liquid in the lower part of the digester wherefrom the partly sulfonated chips are discharged. This is essential to prevent reduction and decomposition of the peroxide solution used as bleaching agent and to avoid formation of sulfuric acid which promotes precipitation of extractive substances on the fibers of the pulp, thus lowering the brightness and absorption capacity of the final product.

The partly sulfonated chips are fed out from the digester by means of a screw conveyor 12. In a preferred embodiment of the invention, the screw conveyor displaces the chips into a disc refiner 13 or similar defibrating device, e.g. a screw defibrating device for high consistency treatment, which provides a kneading and shearing action without rupturing the fibers. Devices of the last-mentioned type are sold by *MdDoMekan AB* under the trade name FROTAPULPER and are identical with the device disclosed in the Swedish patent 314 288. The mechanical defibration is suitably carried out under a pressure equal to or slightly lower than that prevailing in the digester. It is essential according to the invention that in the mechanical defibrating step the cellulose material is defibrated only in part, which means that after the defibrating step the pulp contains fiber bundles as defined above each comprising 2 to 4 individual fibers, which is very advantageous for the bulk of the final product. According to the invention, the partial mechanical defibration should be carried out so that the defibrated pulp contains at least 10 %, preferably 15 to 30 % by weight of the fiber bundles. Further, any type of fiber shortening is preferably avoided during the defibrating step, which may be achieved by treatment in high consistency mills of the disc refiner type or in the above mentioned devices sold under the trade name FROTAPULPER. The partial sulfonation also prevents formation of hard fiber bundles, so-called shives, which are always formed in an amount greater than 2% of the total fiber material when mechanical pulp is manufactured the conventional way.

Resin-removing and bleaching chemicals are added before entry of the chips into the defibrating device 13 through the inlet 11 to the screw conveyor 12. The amount of resin-removing agent added e.g.  $\text{Na}_2\text{PO}_4$ , may for example be from 0.1 to 1.5 % of the pulp weight. Preferably, 0.2 to 0.4 % is used. The charge of surfactants (detergents) is preferably

maximized to 0.2% while the charge of complexing agent such as nitrilotriacetic acid (NTA), ethylenediaminetetracetic acid (EDTA) or diethylenetriamine pentaacetic acid (DTPA) should preferably be at most 0.3 % by weight of the pulp. When hydrogen peroxide is added, the addition should preferably be at most 4% by weight of the pulp, preferably 1.0 to 2.0%. The amount of solution hydroxide added is preferably at least 65 % of the peroxide charge and not above 200 % of the peroxide charge. To stabilize the peroxide, 0.5 % to 4.0 % sodium silicate and/or 0.05 to 0.5 % of magnesium sulfate can be added. When chemicals are to be recovered from recycled water from the process, the addition of sodium silicate should suitably not exceed 0.5 %.

The defibrated cellulose material is blown through the conduit 14 to a cyclone 15 for separating the low pressure steam 17 from the cellulose fibers. The cyclone is provided with a valve 16 to control the discharge of steam and to control the pressure of the steam. The cyclone is combined with a press 18 where the pulp leaving the cyclone is collected and dewatered. The pressure of steam 17 discharged from the cyclone is somewhat below the pressure prevailing in the working space of the disc refiner. The steam can be used for various purposes, e.g. for heating localities or for evaporating liquids containing chemicals. In the press 18 the defibrated cellulose material is dewatered to a solids content of at least 30 %, preferably 45 to 55 %, at an elevated temperature. The effluent liquid leaving the press through an outlet 19 contains useful chemicals and dissolved organic substances, which are suitably recovered by evaporation, separation of extractive substances and combustion or by any other suitable method of recovering the chemicals. Since defibration is generally effected at a pulp consistency of 20 to 40 %, a small volume of excess liquid is obtained. The pulp consistency is reduced during the treatment in the refiner because sealing water has to be introduced.

After passage through the cyclone and the press, the pulp may be passed through another cyclone and press, if desired, to separate steam and to increase the solids content of the pulp, respectively.

The cellulose material is passed after the dewatering step directly, without any intermediate treatment, to a drying installation wherein the moisture content of the cellulose material is reduced to less than 14 %, preferably less than 10 %. The drying installation may comprise a drying machine provided with a wire screen and rolls or a flake dryer. In the first mentioned case, however, the cellulose material must first be diluted to a suitable consistency with recirculating liquid.

The dried pulp produced preferably has a pH of above 6. Most preferably the pH is 7 to 10 so that it has a neutral or slightly alkaline reaction.

The present invention is not limited to the embodiments described above. Thus, for example, the partially sulfonated material may be passed directly from the digester to a pressurized cyclone and may thereupon be defibrated while adding bleaching and resin-removing chemicals and then squeezed. Also, liquid phase digestion may be used instead of vapor phase digestion.

The ratio between the proportion of long-fiber material in a pulp produced in accordance with the invention and the proportion of long-fiber material in a thermomechanical pulp produced in the conventional manner and having a Canadian Standard freeness of 240 ml is seen from a fiber fractionation analysis carried out in a Bauer-McNett apparatus as tabulated below.

Mesh No. (ASTM E11-61)	Pulp of invention		Thermomechanical pulp	
	Retained %	Through %	Retained %	Through %
20	40	60	10	90
150	43	12	60	30

Thus, it will be seen that the pulp produced in accordance with the invention contained about 4 times as much long-fiber materials as the thermomechanical pulp and was close to the fiber composition of chemical pulp. It is also surprising, as will be shown below, that the rate of absorption and the capacity of retaining absorbed water of the pulp produced according to the invention are very good in spite of a relatively high freeness, namely about 450 ml Canadian Standard Freeness. The high fiber length of the pulp produced in accordance with the invention also results in a considerably improved storage stability of absorbent products produced therefrom as compared to thermomechanical pulp. The latter contains so high a proportion of short fibers, that when used in absorbent products it strati-

fies during the storage period, and tends to make the product useless for its intended purpose.

As will be seen from the tests described below the pulp produced according to the invention is exceedingly well suited for use as an absorbent material in products such as diapers, sanitary napkins, tampons and bandaging material. Because of its low resin content it may also be worked up for use as paper-making pulp. In the tests, completely bleached sulfite pulp, peroxide-bleached groundwood and peroxide-bleached thermomechanical pulp were used as reference pulps. The reference pulps were dried to a solids content of 94 % and then conditional to bring the moisture content to 10% after 2 days. The samples were then dry-defibrated (fluffed) in a disc refiner having 300 mm disc diameter. The fluffed pulps were tested with respect to water absorption and bulk. The following testing method was used. Sample pads of 50mm diameter and 2.0m g weight were formed from the fluffed pulp in a special apparatus. By loading the sample with a weight of 100 g and measuring the thickness of the sample a measure of the bulk was obtained. The thickness of the sample was fixed to 15 mm, and it was allowed to absorb water of 293°K from below, the time of absorption being measured. The absorption capacity was determined at a loading of 1000 g.

The results of the tests on the reference pulps are seen from Table I.

TABLE I

		Yield %	Dichloro- methane Extract % SCAN-C 7:62	Brightness % SCAN -C 11:62	Bulk M <sup>3</sup> /kg	Absorp. rate sec.	Absorp. g. water per g. pulp
	Sulfite pulp	52	0.40	92	0.028	26	10.9
	Groundwood	96	1.05	72	0.015	19	10.7
	Thermomech. pulp	95	1.02	71	0.017	9	10.9

A high bulk and a short absorption time indicate that the fluffed pulp is a good raw material for making diapers, for example. As seen from Table I, the sulfite pulp has a high bulk, but a relatively long absorption time. The groundwood has low bulk and a relatively long absorption time. The thermomechanical pulp has low bulk, but short absorption time. The capacity of retaining absorbed water is approximately the same for the samples and may be considered quite acceptable.

The invention is illustrated by the following Example.

*Example*

Two pulps, designated A and B, were prepared of which pulp B was prepared entirely in accordance with this invention. Spruce wood was cut in a chipper to chips having a length of 30 to 50 mm, a width of 10 to 20 mm and a thickness of 1 to 2 mm. The chips were washed in hot water and charged into a steaming vessel, wherein they were heated with saturated steam of atmospheric pressure for 10 minutes. The steamed chips were conveyed by a screw conveyor to an impregnating chamber. The impregnating chamber was filled with a sulfite solution the pH of which was 7.5. The SO<sub>2</sub> content was 5 g/l, and the NaOH content was 6.5 g/l. In the impregnating treatment, the chips absorbed an average of 1.1 liter of sulfite solution per kilogram of chips. Thus, the amount of absorbed SO<sub>2</sub> was 5.5 g per kilogram of chips or 0.55 %. The temperature in the impregnating chamber was maintained at 398°K. The total residence time of the chips in the impregnating chamber was about 2 minutes. During this time, a slight sulfonation of the wood material in the liquid phase was obtained. The impregnated chips were passed to a digester, into which saturated steam was introduced to raise the temperature to 398°K. The residence time of the chips in the digester was 4 minutes. Thus, including the residence time in the impregnating chamber, the total sulfonating time was 6 minutes. From the bottom of the digester, the chips were passed through a screw conveyor to a disc refiner, which was under a steam pressure of 150 kPa (1.5 atmospheres) above atmosphere.

In the disc refiner, the chips were partially defibrated, i.e. a certain proportion of fiber bundles as defined above of 2 to 4 fibers could be found after defibration. The solids content in the center of the disc refiner was 30%, while the pulp consistency at the circumference of the discs was 32 %. The energy input during defibration was 750 kWh per 1000 kg of dry pulp produced. At its exit from the working space of the refiner the pulp was diluted

with sealing water to a pulp consistency of 27 %. The defibrated pulp was blown to a pressurized cyclone to separate excess steam from the pulp fibers. The fibers were collected in a press combined with the cyclone. The press dewatered the pulp from 27 % pulp consistency to 42 % pulp consistency at a temperature of 363°K. Thus, the amount of liquid squeezed out, was only 1.3m<sup>3</sup> per 1000 kg of dry pulp. The amount of organic substances in the solution was 60 kg or 43 g/l, while no free or combined sulfur dioxide could be found. The pulp yield was 94%. The pulp of 42% solids was dried in a flake dryer to a solids content of 94%, conditioned and dry-defibrated and tested in the same manner as the reference pulps above. The pulp thus treated is designated pulp A.

In another experiment, carried out similarly to the preparation of pulp A, resin-removing and bleaching chemicals were added at the center of the grinding discs of the refiner, the following chemicals being charged (expressed as pure chemicals in percent by weight of the dry pulp):

0.1 % EDTA (ethylenediaminetetraacetic acid)

0.3 % Na<sub>2</sub>P<sub>2</sub>O<sub>7</sub>

0.05 % berocell -25 (wetting agent)

2.0 % H<sub>2</sub>O<sub>2</sub>

1.8 % NaOH

2.0 % sodium silicate (water glass) 40°Be

After defibration and dewatering in accordance with the preparation of pulp A, the pulp which was designated pulp B was dried as described above and tested similarly to the reference pulps and pulp A. The results are tabulated in Table II.

TABLE II

Pulps A and B

		Dichloro- methane Extract %	Brightness % SCAN -C 11:62	Bulk M <sup>3</sup> /kg	Absorp. rate sec.	Absorp. g. water per g. pulp
	Yield %	SCAN-C 7:62				
Pulp A	94	1.52	59	0.031	100	5.2
Pulp B	94	0.58	71	0.030	8	11.3

As seen from Table II, both pulp A and pulp B have a surprisssingly high bulk, which is even higher than that of the sulfite pulp exhibited in Table I. As compared to mechanical pulps, the bulk of the pulps produced in accordance with the invention is about twice as high. Pulp A, although it is partially sulfonated, shows a rather poor rate of water absorption.

Upon the addition of bleaching and resin-removing chemicals, however, a surprisingly good rate of absorption and a good capacity of retaining absorbed water have been obtained, and pulp B is superior to the reference pulps and pulp A as an absorption material is absorbent products.

A comparison of the consumption of wood and energy for conventional pulps and pulps according to the invention shows the marked advantages of the present invention.

Wood and energy consumption  
for 1000 kg, dry pulp

	Yield %	Wood m <sup>3</sup>	Energy kWh
Sulfite pulp	52	5.01	800
Groundwood	96	2.71	1400
Thermomech. pulp	95	2.74	2100
Pulps A and B	94	2.77	750

Thus, the process of this invention requires a small energy input and has a low consumption of wood.

Considering furthermore the lowered capital investment for producing pulp according to the invention, because of the elimination of the screening equipment, it will be clear that the method of the invention enables the production of a high grade pulp for absorbent products at a considerably lower cost than has been possible previously and at the same time the discharge to the environment of harmful substances is reduced, since the possibilities of providing a high degree of purification of the waste are improved.

To achieve special effects, the pulp produced according to the invention can be mixed with chemical pulp such that the mixture contains 5 to 30 % by weight of chemical pulp.

#### WHAT WE CLAIM IS:-

1. A light coloured cellulose pulp have a low resin content and high bulk, containing 0.1 to 1.0% by weight of sulfur; a proportion of retained fibers above 25% by weight on a 20 mesh screen (ASTM E 11-61), as determined by a fiber fractionation analysis (as hereinbefore defined) using a Bauer-McNett apparatus; a proportion of soft, sulfonated fiber bundles, each consisting of 2 to 4 individual fibers, of at least 10% by weight, a resin content of less than 0.8% by weight as determined by a dichloromethane extraction procedure according to SCAN-C7:62 and a bulk when dried of above  $20 \times 10^{-3} \text{ m}^3/\text{kg}$ , all percentages being based on the weight of oven dried pulp.
2. A pulp according to claim 1, which contains 0.2 to 0.6% sulfur, has 30 to 65% of its fibers retained on the 20 mesh screen (ASTM E 11-61) as determined by the fiber fractionation analysis using the Bauer-McNett apparatus, contains 15 to 30% soft, sulfonated fiber bundles each containing 2 to 4 individual fibers and has a bulk when dried of above  $25 \times 10^{-3} \text{ m}^3/\text{kg}$ .
3. A pulp according to claim 1 or 2, which has a pH above 6, when dried.
4. A pulp according to claim 3 which has a pH of 7 to 10 when dried.
5. A pulp according to any one of the preceding claims mixed with chemical pulp such that the mixture contains 5 to 30% by weight chemical pulp.
6. A pulp according to claim 1 substantially as hereinbefore described with reference to the Example.
7. A process for making a light-coloured cellulose pulp by washing lignocellulosic material with water, impregnating it with a digesting liquid containing sulfur dioxide-containing digesting chemicals, partly digesting the impregnated lignocellulosic material to a degree of sulfonation corresponding to 0.1 to 1.0% by weight sulfur, based on the weight of the treated cellulose material, at a temperature of 373° to 443°K so that at the termination of the partial digestion, the digesting liquid contains no free or combined sulfur dioxide, mechanically partly defibrating and simultaneously bleaching the lignocellulosic material, the resulting defibrated pulp containing at least 10% by weight, based on the weight of oven dried pulp, of soft, sulfonated fiber bundles each comprising 2 to 4 individual fibers.
8. A process according to claim 7 wherein the partial digestion is carried out to a sulfonation degree corresponding to 0.2 to 0.6% sulfur.
9. A process according to claim 7 or 8 wherein the amount of digesting chemicals in the digesting liquid is at most equivalent to 20 grams of  $\text{SO}_2$  per litre.
10. A process according to any one of claims 7 to 9 wherein the lignocellulosic material is in the form of wood chips.
11. A process according to any one of claims 7 to 10 wherein the defibration is carried out so that after the defibration the defibrated pulp contains 15 to 30% of the soft, sulfonated fiber bundles.
12. A process according to any one of claims 7 to 11 wherein the defibration is carried out in the presence of a resin-removing chemical and a bleaching chemical at an initial pH above 9.
13. A process according to claim 12 wherein the initial pH is above 10.
14. A process according to claim 12 or 13 wherein the resin-removing chemical is a phosphate and the bleaching chemical is an alkali metal peroxide or hydrogen peroxide.
15. A process according to any one of claims 7 to 14 wherein the defibration is followed immediately by dewatering to a solids content of at least 30% by weight at a temperature of at least 348°K.
16. A process according to any one of claims 7 to 14 wherein the defibration is immediately followed by separation of steam, the heat content of the steam being recovered, and by dewatering to a solids content of at least 30% by weight at a temperature of at least 348°K.
17. A process according to claim 15 or 16 wherein the dewatered pulp is dried to a solids content of at least 86%.
18. A process according to claim 17 wherein the dewatered pulp is dried to a solids content of 88 to 92%.



19. A process according to any one of claims 7 to 18 wherein the washed lignocellulosic material is moistened with steam prior to being impregnated with the digesting liquid.

5 20. A process according to any one of claims 7 to 19 wherein the digesting chemical is a bisulfite and/or sulfite of sodium, potassium, ammonium, calcium or magnesium, the total amount of  $\text{SO}_2$  in the digesting liquid being at most equivalent to 20 grams per liter and the pH of the digesting liquid being 5 to 11. 5

21. A process according to claim 20 wherein the pH of the digesting liquid is 6 to 9.

10 22. A process according to any one of claims 7 to 21 wherein the digesting time and temperature are 0.5 to 30 minutes and  $383^\circ$  to  $403^\circ\text{K}$ , respectively. 10

23. A process according to any one of claims 7 to 21 wherein the impregnation with the digesting liquid is effected at a temperature of  $373^\circ$  to  $443^\circ\text{K}$ , for a period of 0.5 to 5 minutes. 10

24. A process according to claim 23 wherein the temperature is  $383^\circ$  to  $403^\circ\text{K}$ .

15 25. A process of making pulp according to claim 7 substantially as hereinbefore described with reference to the Example. 15

26. A process of making pulp according to claim 7 substantially as hereinbefore described with reference to the Figure of the accompanying drawings.

27. Pulp obtained by a process as claimed in any one of claims 7 to 26.

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COMPLETE SPECIFICATION

1 SHEET

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